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Formulation of Ames 24E2 IR-Black Coating

Sheldon M. Smith

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AMES 24E2 IR-BLACK COATING (NASA)
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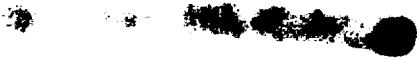
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SUMMARY

The formulation of Ames 24E2 IR-black coating and a rationale for the selection of its components are given. The objective was to make a very rough, very thick, and highly absorbing coating to attenuate the specular reflectance of telescope baffles at far-infrared wavelengths. Application and curing instructions are also given. Outgassing measurements are quite low following a 24-hr radiative cure.

INTRODUCTION

Ames 24E2 IR-black coating was designed to reduce stray light inside far-infrared space telescopes such as SIRTf, COBE, and ISO. Good, but not maximum, stray light reduction at visible and IR wavelengths was desired as well. Attenuation of specular reflection is the principal objective of a "black" coating because, in nearly all telescope designs, if non-imaged or otherwise unwanted light is strongly absorbed and/or scattered at its first baffle reflection, it will not then contribute significantly to the stray-light background at the distant focal plane. An earlier analysis of existing optical black coatings produced a reflecting layer model (ref. 1) which indicates that the most direct way to attenuate normal-incidence specular reflectance is to cover the baffle surface with a very thick, very rough, and highly absorbing coating. That is the philosophy which was followed in designing Ames 24E2. The adjectives "very" and "highly" used here are emphasized because the model indicates that the coating roughness, thickness, and absorptance must be large relative to the wavelength of light, and the wavelengths used in these telescopes are on the order of 1/2 mm.

The author is indebted to Mr. W. A. Campbell of the Goddard Flight Test Center for his advice and assistance during the outgassing tests.

HISTORY

The first very rough, highly absorbing coating was made by J. R. Houck (ref. 2), who loaded 3M's Black Velvet Nextel (101-C10) with large particles of SiC grinding grit. That technique created Cornell Black which had a rms roughness of at least 30 μm and a thickness of about 90 μm . It was quite successful, providing the lowest far-infrared specular reflectance studied in 1984 (ref. 1). Unfortunately the 3M Company had stopped producing its Black Velvet Nextel in late 1980. In a 1986 survey (ref. 3), the far-infrared reflectance of some 35 coatings and additives was measured. This survey indicated among other things that the commercially available coatings Chemglaze Z-306 and ECP-2200 were promising candidates to replace Black Velvet Nextel as the binder, or diluent, of a far-infrared coating. It also indicated that carbon black was a very effective far-infrared absorber. The 24th reflectance spectrum measured in that survey had quite low values. It came from a combination of ECP-2200, carbon black, and #80 SiC grit. A similar combination containing Chemglaze instead of ECP produced somewhat lower reflectance levels; however, ECP was selected as the binder material because of its advertised strong adhesion to aluminum and because it did not require

a primer coating. Thus, Ames 24D came about as the 4th (D) variation of the concentrations of ECP-2200, carbon black, and #80 SiC grit. This formulation seemed adequate until a question regarding the adhesion of the large SiC particles was raised by the failure of a Chemglaze coating to hold glass micro-balloons onto the COBE telescope during a cold vibration test. To make a more adhesive coating, silicone adhesive resin at a concentration of 12% by volume was added to the formulation. This produced Ames 24E. Its normal-incidence specular reflectance compares favorably with that of Cornell Black, as shown by the photometric reflectance spectra of figure 1. Both the specular and diffuse reflectance (BRDF) of Ames 24E are reported in reference 4. Subsequently the concentration of resin has been increased to 20% producing the present coating, Ames 24E2. Its cryo-mechanical and adhesive properties have been measured and reported in reference 5.

COMPOSITION

The formulation of Ames 24E2 is given in table 1. It is strongly recommended that the ECP-2200 be very thoroughly stirred (down to the bottom of the can) before using. Just recently (April 1990) the 3M Company has discontinued production of ECP-2200. Fortunately, the Illinois Institute of Technology Research Institute (IITRI) in Chicago has agreed to produce it in support of several research programs. Mr. Richard J. Mell, of their Chemistry Department, is in charge of the ECP effort. As noted earlier, two reasons for selecting ECP-2200 are that it does not require a primer on aluminum and that it is reported to adhere well to aluminum. A third reason is that it contains small silicate particles, which scatter short wavelengths well and absorb strongly (ref. 6) between 8 and 20 μm .

Carbon black (lampblack) is known to absorb strongly in the far-infrared (ref. 7) as well as in the visible. Raven 1255, produced by the Columbian Chemical Corporation, was selected as the carbon black component because it disperses uniformly in many organic solvents, i.e., it does not usually "ball-up".

Dow Corning silicon adhesive resin Q2-7406 was chosen as the additional adhesive component primarily because it has the same diluent, Xylene, as does ECP-2200. Thus, it dissolves well in ECP. A side benefit of this adhesive is that it never really hardens, thus giving Ames 24E2 a very flexible quality at room temperature. A good deal of this flexibility is retained at cryogenic temperatures, as shown in reference 5 by the high level of strain (bend) that the coating can sustain in liquid nitrogen without shattering, cracking, or detaching from an aluminum substrate.

SiC grit can be obtained from any supplier of grinding compound. One reason for using SiC is its strong absorption (ref. 8) between 2.5 and 14 μm , but the main reason is its size. The reflecting layer model (ref. 1) shows that the specular Fresnel reflectance of the upper surface of the coating will be reduced an order of magnitude by exponential scattering loss when $\sigma/\lambda > 0.12$, where σ is the rms roughness of the upper surface, and λ is the wavelength. At a wavelength of 500 μm , this requires $\sigma > 60 \mu\text{m}$. A photomicrograph by Houck (ref. 2) shows that the modal size of #80 SiC grit is about 300 μm in the longest direction and ~150 μm in the orthogonal directions. Assuming the rms value to be 1/3 the peak-to-valley value, and taking the modal size of the grit as the peak-to-valley roughness, this produces estimates of the rms roughness of 50 to 100 μm . Profilometer measurement

of the upper surface of one sample of Ames 24E and of another of Ames 24E2 gave values of the rms roughness of 56 and 63 μm , respectively. This is surprisingly good agreement for such a rough calculation. It may indicate that some SiC grit is oriented nearly perpendicular to the substrate surface. The average thickness of the coating is about 250 μm .

The ingredients in table 1 are to be mixed together in the concentrations shown. It is recommended that a face mask be worn while measuring and mixing the carbon black, and that the mixing be done in an exhaust hood to remove the nauseating Xylene vapor. The shelf-life of the final mixture is 1.5 yr or more under ambient conditions if (1) the ECP-2200 and the Q2-7406 are fresh at the beginning (they both have 1-yr warranted shelf-lives), and (2) the can lid is scrupulously cleaned and closed between uses. Xylene is the preferred diluent and solvent.

Table 1. Composition of Ames 24E2

Ingredient	Manufacturer	Concentration
ECP-2200	IITTRI, Chicago, IL	48% of the total liquid volume
Carbon black (Raven 1255)	Columbian Chemical Corp., Tulsa, OK	4.75% of the <u>weight</u> of the ECP-2200
Silicone resin (Q2-7406 adhesive)	Dow Corning Corp., Midland, MI	20% of the total liquid volume
#80 SiC grit	Any supplier of grinding compound	32% of the total liquid volume

APPLICATION

Although aluminum is the designed substrate for Ames 24E2, it has been successfully applied to galvanized steel and copper as well. The reflecting layer model (ref. 1) indicates that a very rough substrate surface will also reduce specular reflectance. The substrate should first be thoroughly cleaned with acetone to remove all lubricants, oil, paint, and grease. Do not apply any kind of primer to the substrate. Before application the coating must be thoroughly stirred with a stick to disperse the SiC grit uniformly throughout. Stirring and/or age may cause the coating to thicken excessively. If this has happened, dilute it with Xylene to a milkshake-like consistency and apply with a brush. The first coat acts as a primer and begins to distribute the SiC grit over the surface. Dry longer than 30 minutes in a gentle air draft between coats. The second coat is intended to be thick ($\sim 1/3$ mm) and to distribute the grit uniformly across the surface. Apply a third coat only if the second coat appears thin in places or very uneven.

CURING AND OUTGASSING

The outgassing tests were performed by W.A. Campbell at the Goddard Space Flight Center in accordance with ASTM Test Method E595-84 (ref. 9) and as described in NASA Reference Publication 1124 (ref. 10). The outgassing results are given in table 2, where TML means total mass loss and CVCM means collected volatile condensable material.

The data in table 2 show several things. First, even without an elevated temperature cure Ames 24E and Ames 24E2 coatings do not outgas severely (the ASTM acceptable limits are 1% TML and 0.1% CVCM). Second, the uncured (not baked) comparison between free-standing samples of A24E and A24E2 shows a 30% increase in TML for the E2 formulation, which is consistent with the additional amount of silicone resin in Ames 24E2.

Table 2. Outgassing data comparison

Material	Cure	%TML	%CVCM
A24E	None	0.85	0.28
A24E2	None	1.11	0.29
A24E	24 h @ 135°C	0.67	0.16
A24E2	24 h @ 135°C	0.31	0.00
A24E2	48 h @ 280°C	0.30	0.00

However, comparison of the data after a 24-hr bake-out under mild vacuum at 135 °C of free-standing samples of the two coatings shows a very large decrease of both TML and CVCM for A24E2, which is not consistent with an increased amount of resin. Third, comparison of the A24E2 data after a 48-hr, 280 °C bake-out shows little further decrease in outgassing, although the temperature and duration of cure were significantly increased.

The last two comparisons are consistent with a much improved heating technique developed in order to reach the higher cure temperature of 280 °C. In the earlier method, used on A24E, a free-standing sample of the coating was simply pressed down onto a hotplate at 135 °C inside a vacuum chamber. In order to reach 280 °C, free-standing samples of A24E2 were hung between two quartz lamps inside the vacuum chamber. The sample brightness temperature was read by an IR radiometer (IRCON Model 700) through a CaF₂ window in the chamber and the lamp intensity adjusted to reach the desired brightness temperature. The effectiveness of this radiative heating method is clearly demonstrated by the small change in the outgassing data even though both the temperature and duration of cure were doubled. Radiative curing is clearly the technique of choice. With this type of cure, the outgassing of Ames 24E2 easily meets the acceptable maximums of ASTM E 595-84.

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Photometric Spectra

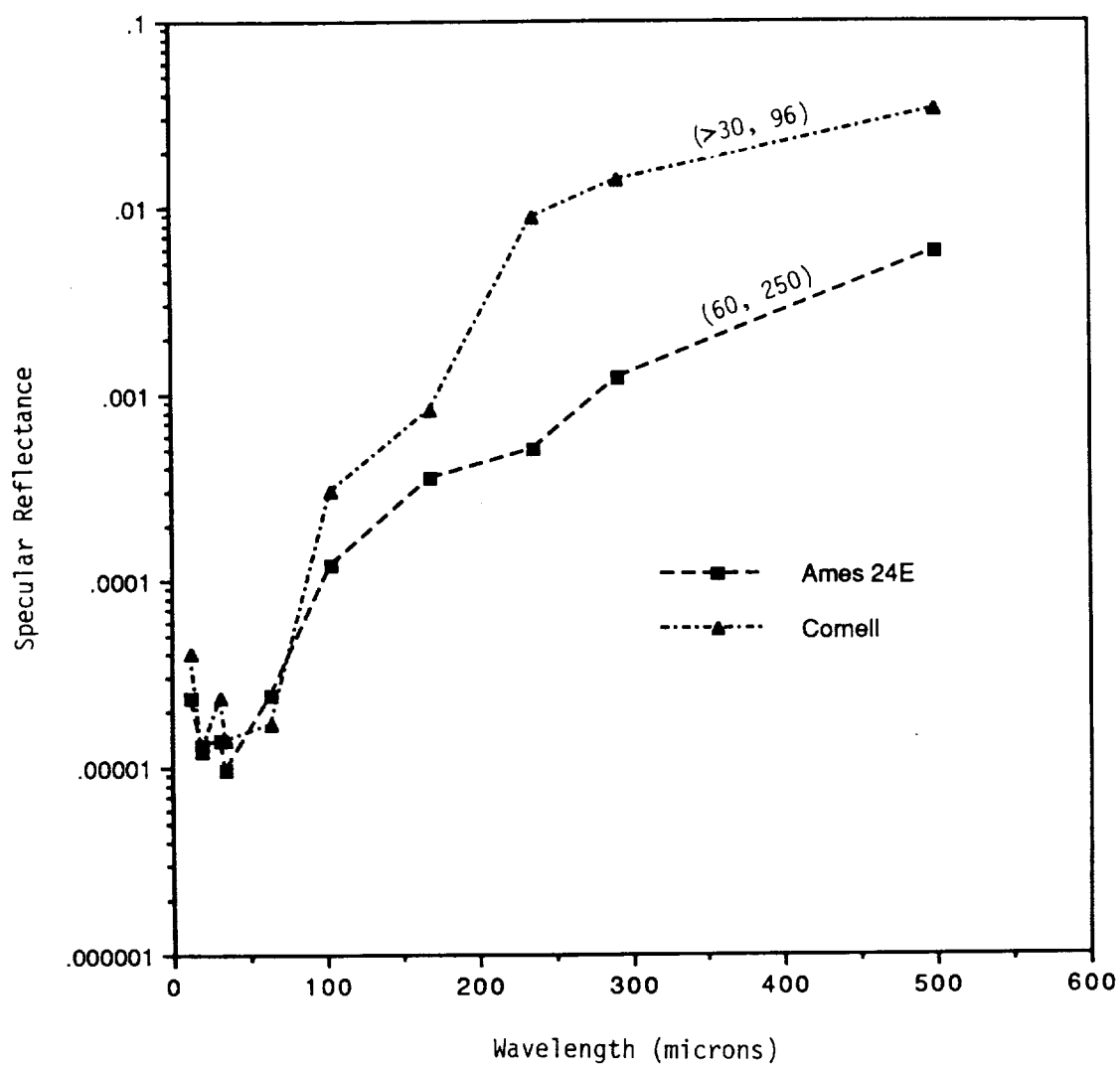


Figure 1. Photometric specular reflectance near normal incidence. Reference 1 contains a detailed discussion of the error of this method of taking spectra. Coating roughness and thickness are given in parentheses (σ , t).

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